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WO 01/34685 A1

(54) Title: OPTIMIZING NANO-FILLER PERFORMANCE IN POLYMERS

(57) Abstract: A polymer composite comprising a polymer matrix having, dispersed therein, a nano clay in combination with a crosslinking promotor. In method form, the invention relates to a process for enhancing the mechanical properties of a polymer composite, comprising supplying a polymer matrix, combining the matrix with a nano clay and a crosslinking promotor and crosslinking.

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OPTIMIZING NANO-FILLER PERFORMANCE IN POLYMERS

The present invention relates to nano clays for use in thermoplastic/thermoset polymer materials, wherein the nano clay may be combined with another chemical ingredient, such as a crosslinking agent, to thereby provide a unique and overall synergistic effect on mechanical property performance.

Polymer composites comprising a polymer matrix having one or more additives such as a particulate or fiber material dispersed throughout the continuous polymer matrix are well known. The additive is often added to enhance one or more properties of the polymer. Useful additives include inorganic layered materials such as talc, clays and mica of micron size.

A number of techniques have been described for dispersing the inorganic layered material into a polymer matrix. It has been suggested to disperse individual layers, e.g., platelets, of the layered inorganic material, throughout the polymer. However, without some additional treatment, the polymer will not infiltrate into the space between the layers of the additive sufficiently and the layers of the layered inorganic material will not be sufficiently uniformly dispersed in the polymer.

To provide a more uniform dispersion, as described in U.S. Pat. No. 4,889,895 sodium or potassium ions normally present in natural forms of mica-type silicates and other multilayered particulate materials are exchanged with organic cations (e.g., alkylammonium ions or suitably functionalized organosilanes) thereby intercalating the individual layers of the multilayered materials, generally by ionic exchange of sodium or potassium ions. This intercalation can render the normally hydrophilic mica-type silicates organophilic and expand its interlayer distance. Subsequently, the layered material (conventionally referred to as "nanofillers") is mixed with a monomer and/or oligomer of the polymer and the monomer or oligomer polymerized.

The intercalated silicate is described as having a layer thickness of 7 to 12 [Angstrom] and an interlayer distance of 30 [Angstrom] or above.

In WO 93/11190, an alternative method for forming a composite is described in which an intercalated layered, particulate material having reactive organosilane compounds is dispersed in a thermoplastic polymer or vulcanizable rubber. Yet additional composites containing these so-called nanofillers and/or their methods of preparation are described in U.S. Pat. Nos. 4,739,007; 4,618,528; 4,528,235; 4,874,728; 4,889,885; 4,810,734; 4,889,885; 4,810,734; and 5,385,776; German Patent 3808623; Japanese Patent J02208358; European Patent applications 0,398,551; 0,358,415; 0,352,042; and 0,398,551; and J. Inclusion Phenomena 5, 473 (1987); Clay Minerals, 23, (1988), 27; Polym. Preprints, 32 (April 1991), 65-66; Polym. Prints, 28, (August 1987), 447-448; and Japan Kokai 76,109,998.

Nano clay fillers are also available based on tiny platelets of a special type of surface modified clay called montmorillonite. These surface treatments have been aimed for use with nylon-6 and polypropylene. The two manufacturers in the United States, Nanocor and Southern Clay Products, both point to increases in flexural modulus, heat distortion temperature and barrier properties.

Furthermore, attention is hereby directed to U.S. Patent Nos. 5,993,415 and 5,998,551 which, although not relating to nano clay fillers, describe the use of crosslinking promotors to improve properties of a thermoplastic material, and, as to be discussed below, are relevant to the present invention. Accordingly, the teachings of these patents are incorporated by reference.

In sum, therefore, even with the numerous described composites and methods, it still remains desirable to have an improved composite and method for forming polymer composites derived from a multilayered additive (nano clays) to thereby create composites having improved properties over the polymer on its own.

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Accordingly, it is an object of this invention to explore the suitability of combining the nano clays with an additional chemical component to establish whether or not the observed mechanical properties of a thermoplastic host resin are improved beyond the use of only a nano clay filler.

More specifically, it is an object of this invention to combine nano clays with a suitable crosslinking promotor, and to establish a synergistic effect of such promotors with the nano clay on the mechanical properties of a host thermoplastic matrix.

In addition, it is an object of this invention to apply the nano clays and additional chemical component described above (promotor) to develop an improved method to prepare materials suitable for use in medical product applications, such as balloon catheters and catheter shaft production.

By way of summary, the present invention comprises a composite comprising a polymer matrix having, dispersed therein, a nano clay in combination with a crosslinking promotor. By use of the term "nano clay" it is noted that such clays are inorganic minerals which have a high aspect ratio with at least one dimension of the particles therein in the nanometer range. By use of the term, "crosslinking promotor" it relates to any chemical compound that will promote crosslinking between those polymer chains that comprise the polymer matrix. Accordingly, it can be appreciate that "crosslinking promotors" include those functionalized chemical compounds that provide the requisite activity, upon activation (irradiation or heat) to chemical react and bond with the polymer chains to form covalent crosslinks between the surrounding polymer chains.

Preferably, the crosslinking promotor is trallylisocyanurate or trallylcyanurate, although those skilled in the art will recognize that other types of crosslinking promotors would be suitable and would fall within the broad aspects of this invention. In addition, preferably, the promotor is

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present in the polymer matrix at a level of about 0.5% to 10% (wt.), and at any increment therebetween in 0.1% increments.

As noted, the nano clays are inorganic minerals with a high aspect ratio as one dimension of the particles therein falls in the nanometer range. A variety of references are available to those skilled in the art which discuss and describe nano clays suitable herein. In such regard, the clays having a plate structure and thickness of less than one nanometer are the clays of choice. The length and width of the clays may fall in the micron range. Aspect ratios of the preferred clays are in the 300:1 to 1,500: 1 range. In addition, the surface area of the exfoliated clays is preferably in the range of 700 m^2/gram. Nano clays that may be suitable herein include hydrotalcite, montmorillonite, mica fluoride, octasilicate, and mixtures thereof. Nano clay is incorporated herein at a level of 1-10% (wt.) as well as any increment therebetween, in 0.1% increments.

Montmorillonite nano clays have a plate like structure with a unit thickness of one nanometer or less. This clay also has an aspect ratio in the 1000:1 range. Because montmorillonite clay is hydrophilic, it is not compatible with most polymers and should be chemically modified to make its surface more hydrophobic. The most widely used surface treatments are amonium cations which can be exchanged for existing cations already on the surface of the clay. The treated clay is then preferably incorported into the polymer matrix herein, by melt mixing by extrusion, more preferably, twin-screw extrusion. In addition, at such time, and as noted above, the crosslinking promotor can also be readily combined with the clay during the melt mixing process. Those skilled in the art will therefore recognize that, in general, any type of melt mixing process can be applied to prepare the composites of the present invention, including extrusion, direct injection molding, the use of a two-roll mill, etc.

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With regards to the development of crosslinking herein, as noted, a crosslinking promotor is employed, and preferably, the formulations herein are exposed to irradiation. Preferably, the irradiation dosage is between about 1-20 MR, as well as any numerical value and/or increment therein.

In addition, the polymer matrix herein may be selected from any thermoplastic or thermoset type polymer resin host. A representative thermoplastic resin herein is a nylon resin, a nylon block copolymer, nylon block copolymers containing a polyamide block and an elastomeric block, engineering thermoplastic resins (e.g., polycarbonate, polyesters, polysulphones, polyketones, polyetherimides) as well as commodity type materials (polyethylene, polypropylene, polystyrene, poly(vinylchloride)) including thermoplastic elastomers. Representative thermoset materials include polyurethanes, epoxy polymers, etc.

In method form, the present invention relates to the steps of supplying a polymer matrix, combining said matrix with a nano clay along with a crosslinking promotor. This combination is then preferably exposed to irradiation to develop crosslinking. By the practice of such method, and as can be observed in the various working examples below, a synergistic influence of the promotor has been observed on the ability of the nano clay to improve the mechanical properties of a given polymer matrix. More specifically, in accordance with the invention herein, it has been found that should one combine a given polymer matrix with the nano clay, one will generally observe an increase in mechanical property performance, such as an increase in the flexural modulus. However, it has been found herein that upon incorporation of a crosslinking promotor, the effect of the nano clay is enhanced, in the sense that a synergy is observed as between the promotor and the nano clay on mechanical properties.

As a consequence of all the above, the formulations of the present invention are particularly suitable for the development of an intravascular

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catheter having a tubular shaft comprising a nylon block copolymer and a nano clay filler, including a compound which promotes crosslinking therein, and a soft flexible tubular tip distal of and bonded to said shaft, the improvement comprising irradiation crosslinking said nylon block copolymer of said tubular shaft. The crosslinking is observed to increase the rigidity of the shaft relative to the soft distal tip.

In addition, the present invention also relates to a balloon type catheter having a tubular shaft comprising a nylon block copolymer and a nano clay filler, including a compound which promotes crosslinking therein, the improvement comprising irradiation crosslinking said nylon block copolymer of the balloon section.

Working Examples

First Experiment

The first experiment consisted of mixing the Nanocor 130 TGC clay and the southern Clay Closite 30B with Nylon 6 and with Nylon 6 and 3% TAIC. The Nylon 6 used with Allied's Capron B135 WP.

The flex modulus did increase with the use of both clays as was anticipated. The increase with the use of a crosslinkng promotor was even greater, demonstrating a unique synergy as between the promotor and the nanoclay on mechanical properties. See Table I.

Second Experiment

The second experiment repeated the first experiment except that the Nylon 6 was replaced by PEBAX® 72 durometer polyamide ether block copolymer. In this case just adding the nano clay did not significantly increase the flex modulus. The surprise was the increase in flex modulus when crosslinking promotors, such as TAIC, was added to the PEBAX® and nano clay. The closite 30B shows the most improvement. A second unqiue effect was the increase in flex modulus when the combination was crosslinked. In fact the combination of PEBAX®, Closite (nano clay) and

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TAIC followed by crosslinking more than doubles the flex modulus. See Table II.

Third Experiment

The third experiment was similar to the first experiment noted above except the nylon-6 was replaced by nylon 12, AESNO® from Atochem. The improvements in flex modulus were much like the improvements with the PEBAX® in "Experiment Two", noted above. See Table III.

Fourth Experiment

The fourth experiment was similar to the third experiment noted above, except that nylon-12 was replaced by nylon-11, BMNO® from Atcohem. The improvements in flex modulus were much like the improvements with the PEBAX® in "Experiment Two". See Table IV.

Fifth Experiment

The fifth experiment was similar to the above, except that both low density and high density polyethylene were employed s the polymer matrix. An improvement in flex modulus was again observed due to the combination of nano clay and promotor (3% wt. TAIC). See Table V.

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NYLON 6

TABLE I

NYLON 6	NONE	CAPRON B135W	0	0MR	9,500	150	350,000
NYLON 6	NANO-130TC	#2	5	0MR	9,500	150	450,000
NYLON 6 3% TAIC	NANO-130TC	29C	5	0MR	6,200	75	410,000
NYLON 6 3% TAIC	NANO-130TC	29C	5	5MR	7,200	15	530,000
NYLON 6 3% TAIC	NANO-130TC	29C	5	10MR	9,500	15	550,000
NYLON 6	CLOSITE 30B	291	5	0MR	9,400	140	510,000
NYLON 6 3% TAIC	CLOSITE 30B	29B	5	0MR	13,250	190	430,000
NYLON 6 3% TAIC	CLOSITE 30B	29B	5	5MR	10,300	25	550,000
NYLON 6 3% TAIC	CLOSITE 30B	29B	5	10MR	10,100	25	590,000
NYLON 6 3% TAIC	NONE	#1	0	5MR	9,500	50	380,000

PEBAX 7233

TABLE II

PEBAX	NONE	PEBAX 7233	0	0MR	8,000	250	105,000
PEBAX	CLOSITE 30B	29E	5	0MR	7,600	200	135,000
PEBAX 3%TAIC	CLOSITE 30B	28A	5	0MR	6,500	180	160,000
PEBAX 3%TAIC	CLOSITE 30B	28A	5	5MR	6,500	75	280,000
PEBAX 3%TAIC	CLOSITE 30B	28A	5	10MR	6,500	50	275,000
PEBAX 3%TAIC	NANO-130TC	28D	5	0MR	9,200	300	135,000
PEBAX 3%TAIC	NANO-130TC	28D	5	5MR	8,200	150	200,000
PEBAX 3%TAIC	NANO-130TC	28D	5	10MR	7,800	125	210,000
PEBAX 3%TAIC	NONE	28E	0	5MR	7,900	150	150,000

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NANO'S WITH XL-NYLON

NYLON 12

TABLE III

POLYMER	FILLER	FORMULATION	FILLER %	IRA DOSE	BK-STRESS	%STRAIN	FLX-MOD
NYLON 12	NONE	AESNO	0	0MR	10,000	250	200,000
NYLON 12	CLOSITE 30B	29A	5	0MR	9,000	200	200,000
NYLON 12 3% TAIC	CLOSITE 30B	28B	5	0MR	10,750	175	290,000
NYLON 12 3% TAIC	CLOSITE 30B	28B	5	5MR	10,250	75	410,000
NYLON 12 3% TAIC	CLOSITE 30B	28B	5	10MR	10,100	50	420,000
NYLON 12	NANO-130TC	#8	5	0MR	9,000	200	190,000
NYLON 12 3% TAIC	NANO-130TC	29D	5	0MR	10,200	300	200,000
NYLON 12 3% TAIC	NANO-130TC	29D	5	5MR	9,500	130	260,000
NYLON 12 3% TAIC	NANO-130TC	29D	5	10MR	9,600	125	260,000
NYLON 12 3% TAIC	NONE	29F	0	5MR	8,000	75	220,000

NANO'S WITH XL-NYLON

NYLON 11

TABLE IV

POLYMER	FILLER	FORMULATION	FILLER %	IRA DOSE	BK-STRESS	%STRAIN	FLX-MOD
NYLON 11	NONE	BMNO	0	0MR	10,000	250	170,000
NYLON 11 3%TAIC	CLOSITE 30B	28C	5	0MR	9,400	200	250,000
NYLON 11 3%TAIC	CLOSITE 30B	28C	5	5MR	9,000	125	300,000
NYLON 11 3%TAIC	CLOSITE 30B	28C	5	10MR	8,500	75	350,000

TABLE V

NANO CLAY IN HDPE & LDPE

POLYMER	FILLER	FORMULATION	FILLER %	IRA DOSE	BK-STRESS	%STRAIN	FLX-MOD	FLX-MOD % INC
HDPE	NONE	3364	0	0MR	2,317	47	73,602	0
HDPE	CLOSITE 30B	30A	6	0MR	2,231	48	81,560	10.8
HDPE	CLOSITE 30B	30A	6	5MR	1,734	32	94,853	28.9
HDPE	CLOSITE 30B	30A	6	10MR	2,474	29	105,069	42.8
HDPE	CLOSITE 30B	30A	6	15MR	2,866	28	111,026	50.8
HDPE	CLOSITE 30B	30A	6	20MR	3,160	28	113,733	54.5
LDPE	NONE	6005	0	0MR	1,018	49	21,295	0
LDPE	CLOSITE 30B	30B	6	0MR	1,000	46	25,856	21.4
LDPE	CLOSITE 30B	30B	6	5MR	1,434	33	29,339	37.8
LDPE	CLOSITE 30B	30B	6	10MR	1,890	35	31,987	50.2
LDPE	CLOSITE 30B	30B	6	15MR	2085	38	31,688	48.8
LDPE	CLOSITE 30B	30B	6	20MR	2,034	30	32,864	54.3

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What is claimed is:

1. A polymer composite comprising a polymer matrix having, dispersed therein, a nano clay in combination with a crosslinking promotor.

2. The composite of claim 1, wherein said crosslinking promotor is a chemical compound which promotes crosslinking between polymer chains upon exposure to irradiation.

3. The composite of claim 1 wherein said crosslinking promotor is triallylisocyanurate or triallylcyanurate.

4. The composite of claim 1 wherein said crosslinking promotor is present at a level of about 0.5 to 10 % (wt).

5. The composite of claim 1 wherein said nano clay is present at a level of about 1 to 10 % (wt).

6. The composite of claim 1 wherein said polymer matrix is a thermoplastic polymer or thermoset polymer.

7. The composite of claim 1 wherein said polymer matrix is a polyamide polymer or a polyamide copolymer comprising a polyamide block and an elastomeric block.

8. The composite of claim 7 wherein said polyamide block is a nylon-6, nylon-6,6, nylon-11, nylon-12, copolymers of nylon-6/nylon-11, copolymers of nylon-6/nylon-12 or mixtures thereof.

9. The composite of claim 7 wherein said elastomeric block is selected from a polyether, polyester, hydrocarbon, polysiloxane or mixtures thereof.

10. A composite comprising a polymer matrix having, dispersed therein, a nano clay in combination with a crosslinking promotor, wherein said matrix is irradiation crosslinked.

11. A method for enhancing the mechanical properties of a polymer composite, comprising:

(a) supplying a polymer matrix;

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(b) combining said matrix with a nano clay and a crosslinking promotor; and

(c) irradiating the combination of step (b) and crosslinking.

12. The method of claim 11 wherein said polymer matrix comprises polymer chains, said promotor is a chemical compound that absorbs irradiation and becomes chemically reactive to form crosslinks, and wherein said crosslinks comprise covalent bonds between said polymer chains.

13. The method of claim 12, wherein said irradiation is 5, 10, 15 or 20 megarads.

14. In an intravascular catheter having a tubular shaft comprising a nylon block copolymer and a soft flexible tubular tip distal of and bonded to said shaft, the improvement comprising adding a nano clay filler and a compound which promotes crosslinking therein to said nylon block copolymer forming said shaft, and irradiation crosslinking said nylon block copolymer of said tubular shaft.

15. In a balloon type catheter having a tubular shaft comprising a nylon block copolymer and an integrally formed balloon section, the improvement comprising adding a nano clay filler and a compound which promotes crosslinking therein to said nylon block copolymer forming said balloon, and irradiation crosslinking said nylon block copolymer of the balloon section.

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/31174

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : C08J 3/28, 9/00, 9/06; C08K 3/34, 3/20, 3/22; C08L 23/06.
US CL : 522/83, 117, 137; 523/216, 300, 521; 524/445, 447, 449, 451; 604/96, 508, 523.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
U.S. : 552/83, 117, 137; 523/216, 300, 521; 524/445, 447, 449, 451; 604/96, 508, 523.

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
Please See Continuation Sheet

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 4,444,816 A (RICHARDS et al) 24 April 1984, Abstract, column 1, line 43, to column 2, line 66, column 3, lines 39-45, and Table 3.	1-15
Y	US 4,385,136 A (ANCKER et al) 24 May 1983, column 6, line 26, to column 7, line 49, column 8, lines 5-27, column 10, lines 26-37, Table II, column 11, lines 30-40, column 16, line 30, to column 17, line 10, Examples 1 and 3-6.	1-15
X --- Y	US 5,853,886 A (PINNAVAIA et al) 29 December 1998, Abstract, column 3, line 49, to column 4, line 6, column 6, line 13, to column 8, line 18, column 9, lines 4-10, column 9, lines 60-66, column 10, lines 16-26 and lines 57-65, column 13, line 64, to column 14, line 4, column 14, lines 37-47, Examples E4, E13 and E14.	1,5,6,7,10 --------------- 4,11-13
X --- Y	WO 93/11190 (ALLIED-SIGNAL, INC.) 10 June 1993, Abstract, page 7, lines 8-15, column 8, lines 14-20, column 10, lines 26-30, column 21, lines 23-29, pages 23-25, page 28, lines 8-16, page 30, lines 23-29, page 33, line 28, to page 34, line 19, and Example 1.	1,5-8,10 ------------ 4,11-15
A	US 4,303,595 A (ALLEN) 01 December 1981, Abstract, column 2, lines 56-61, column 3, line 61, to column 4, line 22.	14-15
Y,P	US 5,993,415 A (O,NEIL et al) 30 November 1999, Abstract, column 3, lines 37-58, and column 4, lines 37-50.	1-15

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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Date of the actual completion of the international search	Date of mailing of the international search report 26 FEB 2001
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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/31174

C (Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y,P	US 5,994,445 A (KASHEL et al) 30 November 1999, Abstract, column 3, lines 14-34, column 3, line 53, to column 4, line 27.	1-15
Y,P	US 6,034,163 A (BARBEE et al) 07 March 2000, column 2, lines 22-44, column 3, lines 12-35, column 6, lines 35-61, column 7, lines 52-55.	1-15
Y,P	US 6,136,908 A (LIAO et al) 24 October 2000, Abstract and column 2.	1-15

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Continuation of B. FIELDS SEARCHED Item3: USPAT, DERWENT, EPO, JPO: nano clay, nano fillers, nano composites, montmorillonite, hydrotalcite, mica fluoride, ostasilicate, clay, talc, mica, silicate, intercalate, nylon, polyamide, polyethylene, block copolymer